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(54) Title: PERFUME COMPOSITIONS

(57) Abstract

A perfume composition contains at least 15 % by weight one or more defined aldehydes, and at least 30 % by weight of at least one of five defined categories of hydroxylic materials, ketones, ethers, esters and nitriles. The perfume composition exhibits a deodorant effect, is particularly good at reducing sulphurous malodours, and is suitable for use in deodorant products, personal products and laundry treatment products.

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PERFUME COMPOSITIONS

This invention relates to perfume compositions, that is to say compositions of fragrance materials, to personal products and other products containing such perfume compositions, and to the use of such perfume compositions to give a deodorant effect.

5 EP-B-3172, EP-A-5618, US-A-4304679, US-A-4322308, US-A-4278658, US-A-4134838. US-A-4288341, USA-4289641 and US 4 906 454 all describe perfume compositions which exhibit a deodorant action, (i.e. addresses the problem of human body odour, particularly that of the axillæ) either when applied to human skin using a cosmetically acceptable vehicle or when included in a detergent product or fabric conditioning product used in laundering of
10 textiles. EP-B-147191 and US-A-4663068 describes deodorant perfume compositions which are stable in the presence of bleaching materials.

A difficulty with the perfume compositions disclosed in these documents is that they generally include appreciable quantities of relatively high molecular weight perfume components which help to extend the effective lifetime of deodorant action following product application, but
15 which tend to have less perfume impact and to exhibit odour characteristics which span a finite range. This limitation on perfume composition represents a compromise between long term deodorant efficacy and optimal hedonic performance.

We have now found that deodorant perfumes can be made by the use of materials from certain specified categories of perfume materials which makes it possible to obtain fragrances
20 containing lower quantities of high boiling components while also obtaining good long term deodorant properties. Forms of this invention can deliver a deodorant performance which improves on that obtained from compositions exemplified in the prior documents above. In particular compositions according to the invention are good at reducing sulphurous malodours in body odour.

25 Accordingly, the present invention provides a perfume composition comprising

- (1) at least 15% by weight of the perfume composition of one or more aldehydes of general formula



having an octanol-water partition coefficient within the range of 2.0 to 4.4 (in logarithmic form),
30 and wherein the group R' is a hydrocarbyl radical which may comprise aromatic or aliphatic groups, and mixtures thereof, and which may be cyclic or acyclic, straight chained or branched, and optionally substituted with other groups, with the proviso that at least 1% by weight of the perfume composition comprises aliphatic aldehydes of this general formula, and
35 (2) at least 30%, preferably at least 70%, by weight of the perfume composition of at least one, preferably at least two, and more preferably at least three of the following five categories of perfumery ingredients;

a) at least 2.0%, preferably at least 4.0%, by weight of the perfume composition of one or more hydroxylic materials of general formula



5 having an octanol-water partition coefficient within the range of 2.5 to 3.6 (in logarithmic form), and wherein the group R' is a hydrocarbyl radical containing no more than one olefinic double bond, and comprising aromatic or aliphatic groups, and mixtures thereof, and which may be cyclic or acyclic, straight chained or branched, and optionally substituted with other groups,

10 b) at least 2.0%, preferably at least 4.0%, by weight of the perfume composition of one or more ketones of general formula



15 having an octanol-water partition coefficient within the range of 3.0 to 4.1 (in logarithmic form), and wherein the groups R' and R² are independently hydrocarbyl radicals which may comprise aromatic or aliphatic groups, and mixtures thereof, and may be cyclic or acyclic, straight chained or branched, and optionally substituted with other groups,

c) at least 2.0%, preferably at least 4.0%, by weight of the perfume composition of one or more ethers of general formula



20 having an octanol-water partition coefficient within the range 3.0 to 4.0 (in logarithmic form), and wherein the groups R' and R² are independently hydrocarbyl radicals which may comprise aliphatic or aromatic groups, and mixtures thereof, and which may be straight chained or branched and optionally substituted with other groups, with the proviso that at least one of R' and R² comprises an olefinic double bond,

25 d) at least 2.0%, preferably at least 4.0%, by weight of the perfume composition of one or more esters of general formula



30 having an octanol-water partition coefficient within the range 2.6 to 4.3 (in logarithmic form), and wherein the groups R' and R² are independently hydrocarbyl radicals which may comprise saturated aliphatic or aromatic groups, and mixtures thereof, and which may be straight chained or branched, cyclic or acyclic, and optionally substituted with other groups, and

e) at least 2.0%, preferably at least 4.0% by weight of the perfume composition of one or more nitriles of general formula



35 having an octanol-water partition coefficient within the range 3.0 to 4.4 (in logarithmic form), and wherein the group R' is a hydrocarbyl radical comprising an olefinic double bond, which may comprise aliphatic or aromatic groups, and mixtures thereof, and which may be straight chained or branched, cyclic or acyclic and optionally substituted with other groups.

The invention also provides a deodorant product comprising a perfume composition as defined above.

The invention further provides the use, as a deodorant, of a perfume composition and a deodorant product as defined above.

5 The term 'perfume material (or ingredient)' is herein taken to represent materials which may be acceptably employed within fragrances to provide an odour contribution to the overall hedonic performance of the fragrance. Typically, such materials will be generally recognised as possessing odours in their own right, and will be relatively volatile, and characterised by molecular weights within the range of around 100 to 300 amu.

10 The concentration of perfume materials or ingredients referred to herein is relative to the total concentration of perfume components present in the composition, ie excludes, for example, the presence of any optional diluent.

15 The octanol-water partition coefficient (or its common logarithm to base 10, 'logP') is well known in the literature as an indicator of hydrophobicity and water solubility (see Hansch and Leo, *Chemical Reviews*, 526 to 616, (1971), 71; Hansch, Quinlan and Lawrence, *J.Organic Chemistry*, 347 to 350 (1968), 33). Where such values are not available in the literature they may be measured directly, or approximately estimated using mathematical algorithms. Software providing such estimations are available commercially, for example 'LogP' from Advanced Chemistry Design Inc.(ACD). For the purposes of the present invention the results obtained using ACD software are preferred.²⁰

A perfume composition according to the present invention has the following preferred features, either singly or in any combination;

- (i) hydroxylic materials of category (2) (a) which are one or more of
25 Citronellol
Dimethylheptanol
Tetrahydrolinalol,
(ii) aldehydes of category (1) which comprise less than two hydrogen atoms in the position directly adjacent to the formyl functional group,
(iii) ethers of category (2) (c) in which R¹ comprises an alicyclic or aromatic ring, and
30 (iv) esters of category (2) (d) in which R¹ or R² comprises an alicyclic or aromatic ring.

In cyclic esters according to category (2) (d), R¹ and R² may be directly connected, as in lactones.

In certain instances, it may be that materials are capable of classification into more than one

category, for example, 4-(4'-hydroxy-4'-methylpentyl)cyclohex-3-enecarbaldehyde comprises both hydroxyl and formyl functional groups and hence could be a member of category (1) as well as (2) (a). In such cases the material is deemed to be within the first named category, that is, in (1) before (2), in (2) (a) before (2) (b) and so forth. Acetals are considered herein as ethers. With regard to the essential oils, synthetic oils and complex mixtures common within the perfumery business, the above rules must be applied to their individual constituents.

Preferred perfumes comprise at least 50%, more preferably at least 60%, and particularly at least 75% by weight of perfume ingredients as described herein. The perfume composition preferably comprises at least 2, more preferably at least 3, and particularly at least 4 of the 5 10 classes of perfumery ingredients (2) (a) to (2) (e).

The invention is directed to perfume compositions and to consumer products which provide a deodorant action when applied to the body within a cosmetically acceptable vehicle. Suitable deodorant products include, but are not limited to, deodorants and antiperspirants including different physical forms such as roll ons, gels, sticks, and aerosols, other personal products 15 such as deocolognes, talcum powders, hand creams, lotions, skin and hair conditioners, sunscreens, soaps, shampoos, and shower gels.

The perfumes described herein may also be usefully employed for deodorant properties in other product areas, for example in detergent and household products such as laundry powders, laundry liquids, rinse conditioners, and household cleaning compositions. Perfumes 20 of the inventions may also be incorporated into textiles directly during manufacture using techniques known in the art, to provide long lasting deodorant protection. It is also known in the art to carry or encapsulate perfumes within other materials such as porous solids or polymeric matrices, in order to provide extended lifetimes, and to provide the possibility of triggered release, for example, during perspiration. Such techniques are applicable within the 25 scope of the present invention.

The invention is illustrated by the following examples.

EXAMPLES

Table 1 below presents a representative list of perfume materials falling within the above categories, together with comparative examples of commonly used perfume ingredients which 30 do not fall within the categories.

Tables 2 to 4 illustrate the use of perfumes of the invention within deodorant products. Their preparation is well known to those skilled in the art.

A Sulphur Reduction Test, suitable for determining reduction of sulphurous malodours in body

odour is as follows:

- Take 200 μ l of saturated vapour from above a vial containing equal weights of ethanethiol and methyl sulphide using a gas-tight syringe. Inject the sulphur-containing materials into the headspace volume of a closed glass vial containing 0.25g of perfume or perfume ingredient.
- 5 Allow to equilibrate for 2 minutes, then remove a small aliquot (e.g. 0.5ml from a 20 ml vial) of mixed vapours from the headspace above the perfume material and analyse by gas chromatography. Care is taken throughout to preserve the integrity of both samples and reduce losses or contamination. The perfume/thiol/sulphide mixture is allowed to equilibrate for a further 13 minutes, after which a second aliquot is sampled and analysed as previously.
- 10 The concentration of sulphurous materials in the vial headspace at 2 minutes and 15 minutes is compared. If the concentration has been reduced by 75% or more, the perfume or perfume ingredient is deemed useful for reducing sulphurous compounds.

Table 1

Examples of Ingredient Categorisation

Material	Category
1,3,4,6,7,8-hexahydro-4,6,6,7,8,8-hexamethylcyclopenta[g]-2-benzopyran	ex
1-(3,5,5,6,8,8-hexamethyl-5,6,7,8-tetrahydro-2-naphthalenyl)-1-ethanone	ex
1-Methyl-2-propenyl-4-isopropyl benzene	ex
2 - tert - Butylcyclohexyl acetate	(2) (d)
2,4 - dimethylcyclohex - 3 - ene - 1 - carbaldehyde	(1)
2-Ethyl-4-(2,2,3-trimethyl-3-cyclopenten-1-yl)-2-buten-1-ol	ex
2-methyl-3-(4'-(1"-methylethyl)phenyl)propanal	(1)
4-(4'-hydroxy-4'-methylpentyl)cyclohex-3-enecarbaldehyde	(1)
8,8-Dimethyl-7-(1-methylethyl)-6,10-dioxaspiro[4.5]decane	(2) (c)
Acetyl di-isoamylene	(2) (b)
Benzyl salicylate	ex
Cedrenyl acetate	ex
Citronellol	(2) (a)
Citronellyl nitrile	(2) (e)
Coumarin	ex
Diethyl phthalate	ex
Diethyldimethylcyclohex-2-en-1-one	(2) (b)
Dihydrojasnone	(2) (b)

Dihydrojasmone	(2) (b)
Dimethyl benzyl carbinyl acetate	(2) (d)
Dimethyl hexan-1-ol	(2) (a)
Dipropylene glycol	ex
Florocylene	ex
Heliotropin	ex
Hexyl cinnamic aldehyde	ex
Hexyl salicylate	ex
Ionones	(2) (b)
Methyl ionones	ex
Methyl isoeugenol	(1) (c)
Phenylethyl alcohol	ex
Tetrahydrolinalool	(2) (a)
Undecalactone, gamma-	(2) (d)
Vanillin	ex

Note: 'ex' = excluded from classification

Table 2
Perfume composition

Material	w/w %	category
1,3,4,6,7,8-hexahydro-4,6,6,7,8,8-hexamethylcyclopenta[g]-2-benzopyran	8.9	
2 - tert - Butylcyclohexyl acetate	2	2 (d)
2,4 - dimethylcyclohex - 3 - ene - 1 - carbaldehyde	0.5	1
2,6,10-trimethylundec-9-enal	0.2	
2-methyl-3-(4'-(1"-methylene)phenyl)propanal	4.6	1
8,8-Dimethyl-7-(1-methylethyl)-6,10-dioxaspiro[4.5]decane	6	2 (c)
Acetyl di-isooamylene	5.5	2 (b)
Cassis base AB2967 (Q)	0.6	
Cervolide (Q)	3	
Diethyl phthalate	9	
Diethyldimethylcyclohex-2- n-1-one	3	2 (b)
Dihydrojasmone	0.5	2 (b)
Dimethyl benzyl carbinyl acetate	2	2 (d)
Hexyl cinnamic aldehyde	5.7	

Ionone, alpha-	8.5	2 (b)
Methyl dihydrojasmonate	12	2 (d)
Lyrat™	12	1
Methyl isoeugenol	2	2 (c)
Muguet base AB1951 (Q)	2.5	
Tetrahydrolinalol	11	2 (a)
Undecalactone, gamma-	0.5	2 (d)
total	100%	

- Materials marked 'Q' are available from Quest International
- Lyrat is a mixture of 4-(4'-hydroxy-4'-methylpentyl)cyclohex-3-enecarbaldehyde and 3-(4'-hydroxy-4'-methylpentyl)cyclohex-3-enecarbaldehyde
- The perfume comprises over 70% of ingredients according to the invention

5 The above perfume composition was particularly effective in the Sulphur Reduction Test.

Table 3

Deodorant stick

Ingredient	Weight %
Ethanol	44.0
Sodium Stearate	7.0
Propylene glycol	11.0
Perfume	1.0
PEG-6-Caprylic/capric glycerides	12.0
Glycerin	5.0
Water	20.0

Table 4

Deodorant aerosol

Ingredient	Weight%
Isopropyl myristate	3.0
Propellants	to 100%
Fumed silica	0.25

Perfume	1.5

Table 5

Roll-ons

Ingredient	Weight%	Weight%
Ethanol		60.0
Klucel MF		0.65
Cremophor RM410		0.5
Bentone gel IPM (Rheox Inc.)	27.0	
Silicone fluid DC344 (Dow Corning)	to 100%	
Aluminium chlorhydrate powder	20.0	
Perfume	0.75	1.00
Water		to 100%

CLAIMS

1. A perfume composition comprising

(1) at least 15% by weight of the perfume composition of one or more aldehydes of general formula

5 R^1CHO

having an octanol-water partition coefficient within the range of 2.0 to 4.4 (in logarithmic form), and wherein the group R^1 is a hydrocarbyl radical which may comprise aromatic or aliphatic groups, and mixtures thereof, and which may be cyclic or acyclic, straight chained or branched, and optionally substituted with other groups, with the proviso that at least 1% by

10 weight of the perfume composition comprises aliphatic aldehydes of this general formula, and

(2) at least 30%, preferably at least 70%, by weight of the perfume composition of at least one, preferably at least two, and more preferably at least three of the following five categories of perfumery ingredients:

a) at least 2.0%, preferably at least 4.0%, by weight of the perfume composition of
15 one or more hydroxylic materials of general formula

R^1OH

having an octanol-water partition coefficient within the range of 2.5 to 3.6 (in logarithmic form), and wherein the group R^1 is a hydrocarbyl radical containing no more than one olefinic double bond, and comprising aromatic or aliphatic groups, and mixtures thereof, and which may be cyclic or acyclic, straight chained or branched, and optionally substituted with other groups,
20 b) at least 2.0%, preferably at least 4.0%, by weight of the perfume composition of one or more ketones of general formula

$R^1CO\ R^2$

25 having an octanol-water partition coefficient within the range of 3.0 to 4.1 (in logarithmic form), and wherein the groups R^1 and R^2 are independently hydrocarbyl radicals which may comprise aromatic or aliphatic groups, and mixtures thereof, and may be cyclic or acyclic, straight chained or branched, and optionally substituted with other groups,

30 c) at least 2.0%, preferably at least 4.0%, by weight of the perfume composition of one or more ethers of general formula

$R^1O\ R^2$

35 having an octanol-water partition coefficient within the range 3.0 to 4.0 (in logarithmic form), and wherein the groups R^1 and R^2 are independently hydrocarbyl radicals which may comprise aliphatic or aromatic groups, and mixtures thereof, and which may be straight chained or branched and optionally substituted with other groups, with the proviso that at least one of R^1 and R^2 comprises an olefinic double bond,
d) at least 2.0%, preferably at least 4.0%, by weight of the perfume composition of one or more esters of general formula

40 $R^1CO_2R^2$

having an octanol-water partition coefficient within the range 2.6 to 4.3 (in logarithmic form), and wherein the groups R¹ and R² are independently hydrocarbyl radicals which may comprise saturated aliphatic or aromatic groups, and mixtures thereof, and which may be straight chained or branched, cyclic or acyclic, and optionally substituted with other groups, and

5 e) at least 2.0%, preferably at least 4.0% by weight of the perfume composition of one or more nitriles of general formula



10 having an octanol-water partition coefficient within the range 3.0 to 4.4 (in logarithmic form), and wherein the group R¹ is a hydrocarbyl radical comprising an olefinic double bond, which may comprise aliphatic or aromatic groups, and mixtures thereof, and which may be straight chained or branched, cyclic or acyclic and optionally substituted with other groups.

2. A perfume composition according to claim 1 wherein the hydroxylic materials of
15 category (2) (a) comprise one or more of the following materials

Citronellol

Dimethylheptanol

Tetrahydrolinalol.

3. A perfume composition according to either one of claims 1 and 2 wherein the
20 aldehydes of category (1) comprise less than two hydrogen atoms attached to the carbon atom adjacent to the carbonyl function.

4. A perfume composition according to any one of the preceding claims wherein the ethers of category (c) comprise an alicyclic or aromatic ring within R¹.

5. A perfume composition according to any one of the preceding claims wherein the
25 esters of category (d) comprise an alicyclic or aromatic ring within R¹ or R².

6. A perfume composition according to any one of the preceding claims comprising at least 50% by weight of specified ingredients.

7. A perfume composition according to any one of the preceding claims comprising at least 60% by weight of specified ingredients.

30 8. A perfume composition according to any one of the preceding claims comprising at least 75% by weight of specified ingredients.

9. A deodorant product comprising a perfume composition defined in any one of the preceding claims.

10. A deodorant product according to claim 9 wherein the product is a personal product
35 including shampoos, creams, lotions, conditioners, soaps and talcs.

11. A deodorant product according to claim 9 wherein the product is a laundry treatment product including detergents and rinse conditioners.

12. The use, as a deodorant, of a perfume composition defined in any one of claims 1 to 8, and of a deodorant product defined in any one of claims 9 to 11.

A. CLASSIFICATION OF SUBJECT MATTER

IPC 7 A61K7/46 A61K7/32

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

IPC 7 A61K

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practical, search terms used)

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category *	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	US 3 325 369 A (INT. FLAVORS AND FRAGRANCES, INC.) 13 June 1967 (1967-06-13) column 1, line 11 - line 13 example 1 ---	1-3, 6, 9, 10, 12
X	FR 2 666 510 A (LABO WUEST SA) 13 March 1992 (1992-03-13) page 1, line 24 -page 2, line 24 page 3, line 26 -page 4, line 2 page 4, line 31 - line 35 page 5, line 3 - line 4 ---	1-3, 6-10, 12
X	EP 0 076 493 A (DRAGOCO GERBERDING CO GMBH) 13 April 1983 (1983-04-13) example 5 claims ---	1-3

Further documents are listed in the continuation of box C.

Patent family members are listed in annex.

* Special categories of cited documents :

- "A" document defining the general state of the art which is not considered to be of particular relevance
- "E" earlier document but published on or after the international filing date
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11 November 1999

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Name and mailing address of the ISA

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C.(Continuation) DOCUMENTS CONSIDERED TO BE RELEVANT

Category	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
A	EP 0 834 551 A (HENKEL KGAA) 8 April 1998 (1998-04-08) abstract page 2, line 43 -page 3, line 7 page 3, line 33 - line 38 page 8, line 22 - line 26 table 3 ---	1-3,5-7, 9-12
A	WO 97 30689 A (PROCTER & GAMBLE) 28 August 1997 (1997-08-28) page 3, line 14 - line 32 page 4, line 10 -page 5, line 32 table 1 example M ---	1-3,6,7, 9,10,12
A	EP 0 545 556 A (QUEST INT NEDERLAND) 9 June 1993 (1993-06-09) abstract page 2, line 18 -page 6, line 48 table 1 ---	1-3,5,6, 9-12
A	US 5 733 535 A (HOLLINGSHEAD JUDITH ANN ET AL) 31 March 1998 (1998-03-31) column 2, line 5 - line 9 column 2, line 21 - line 50 column 4, line 56 -column 6, line 14 page 19, line 1 - line 30 -----	1-3,5,9

INTERNATIONAL SEARCH REPORT

International application No.

PCT/GB 99/02163

Box I Observations where certain claims were found unsearchable (Continuation of Item 1 of first sheet)

This International Search Report has not been established in respect of certain claims under Article 17(2)(a) for the following reasons:

1. Claims Nos.: because they relate to subject matter not required to be searched by this Authority, namely:

2. Claims Nos.: because they relate to parts of the International Application that do not comply with the prescribed requirements to such an extent that no meaningful International Search can be carried out, specifically:
see FURTHER INFORMATION sheet PCT/ISA/210

3. Claims Nos.: because they are dependent claims and are not drafted in accordance with the second and third sentences of Rule 6.4(a).

Box II Observations where unity of invention is lacking (Continuation of Item 2 of first sheet)

This International Searching Authority found multiple inventions in this international application, as follows:

1. As all required additional search fees were timely paid by the applicant, this International Search Report covers all searchable claims.

2. As all searchable claims could be searched without effort justifying an additional fee, this Authority did not invite payment of any additional fee.

3. As only some of the required additional search fees were timely paid by the applicant, this International Search Report covers only those claims for which fees were paid, specifically claims Nos.:

4. No required additional search fees were timely paid by the applicant. Consequently, this International Search Report is restricted to the invention first mentioned in the claims; it is covered by claims Nos.:

Remark on Protest

- The additional search fees were accompanied by the applicant's protest.
 No protest accompanied the payment of additional search fees.

FURTHER INFORMATION CONTINUED FROM PCT/ISA/ 210

Continuation of Box I.2

Present claims 1-12 relate to a composition, a product and a use defined by reference to the parameter: "octanol-water partition coefficient". The values of this parameter are only known for a selection of the compounds embraced by the claims. As a consequence, the use of this parameter in the present context is considered to lead to a lack of clarity within the meaning of Article 6 PCT. It is not fully possible to compare the parameter the applicant has chosen to employ with what is set out in the prior art. The lack of clarity is such as to render a meaningful complete search impossible. Moreover, the structural definitions of the compounds are not fully defined, and are open-ended (e.g. "optionally substituted with other groups") leaving doubts as to the question which compounds actually are comprised by the claims, and which are not comprised by the claims. A meaningful search over the whole scope of the claims is virtually impossible in this situation. Consequently, the search has been restricted to the compounds cited in the examples (Tables 1-2, p. 5-7), the compounds for which the ClogP-value, which corresponds to the octanol-water partition coefficient, was given in WO9730689 (Tables 1-2, p. 7-9 and Perfumes A-Q, p. 61-71), obvious variants thereof and the general idea underlying the application.

The applicant's attention is drawn to the fact that claims, or parts of claims, relating to inventions in respect of which no international search report has been established need not be the subject of an international preliminary examination (Rule 66.1(e) PCT). The applicant is advised that the EPO policy when acting as an International Preliminary Examining Authority is normally not to carry out a preliminary examination on matter which has not been searched. This is the case irrespective of whether or not the claims are amended following receipt of the search report or during any Chapter II procedure.

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